Thin Films
Room 303C - Session TF-WeM

Optical Thin Films
Moderator: G. Ockenfuss, OCLI - JDS Uniphase

8:20am **TF-WeM1 Optical Properties of Uranium and Thorium-Based Thin Films as Highly Reflective EUV Mirrors**, *D.D. Allred, J.E. Johnson,* ***W.R. Evans****, N. Farnsworth, A.E. Baker, R.S. Turley,* Brigham Young University

We present measured reflectances (Beamline 6.3.2, ALS at LBNL) of air oxidized sputtered uranium and thorium, reactively sputtered (O2) uranium oxide and thorium, and reactively sputtered (N2) uranium nitride thin films. The thin films were also characterized using AFM, spectroscopic ellipsometry, LAXRD, TEM, XPS, and XANES. We compare these with our measurements of the reflectance of nickel, gold, and iridium thin films (commonly used soft x-ray reflectors for astronomy and synchrotrons) from 50 to 500 eV at 5Ës, 10Ës, and 15Ës grazing incidence. These show that these two uranium compounds, as UO2 and UN, and thorium as air oxidized thorium can fulfill their promise as the highest-known, single-surface reflector for this portion of the soft x-ray region, being nearly twice as reflective as nickel in the 120 to 200 eV region. We additionally report complex indices of refraction for ThO2, UO2, and UN obtained from reflection and transmission measurements taken at the ALS. Shifts in energy of up to 20 eV between features in the Th spectra suggest the need for possible revision in the atomic scattering factor approach for obtaining optical constants for compounds. There are also important discrepancies between UO2â?Ts and UNâ?Ts actual reflectance with those estimated using the atomic scattering factor approach.

8:40am **TF-WeM2 Determining Physical and Chemical Properties of Sputtered Uranium and Thorium Thin Films Useful as Extreme Ultraviolet Reflectors**, ***R.W.L. Larsen****,* Provo High School*; D.D. Allred,* Brigham Young University

As applications for extreme ultraviolet radiation have been identified, the demand for better optics has also increased. Recently, sputtered thin films containing uranium and thorium have been shown to produce the highest low-angle reflectors for portions of the EUV/soft x rays. For more progress to be made in this area optical constants needed to be obtained. In addition, optical properties need to be assigned to specific compounds and structures. The purpose of this project was to determine the composition, density, lattice structure, and roughness of these sputtered uranium and thorium thin films and their oxides and nitrides via TEM, XPS, AFM and to relate these structural effects to the reflectance of the thin films. We have found that the thorium samples oxidize more slowly in air than uranium so we can talk about the reflection of these films being a mixture of thorium and thorium oxide. Sputtered uranium on the other hand quickly becomes uranium dioxide which is moderately stable for periods of months. Whereas the sputtered uranium nitride samples are uranium mononitride only in the bulk and their surfaces become oxidized.

9:00am **TF-WeM3 Mechanical Characteristics of Optical Films: Their Origin and Evaluation**, ***L. Martinu****, J.E. Klemberg-Sapieha,* Ecole Polytechnique Montreal, Canada **INVITED**

Control of the mechanical properties is essential in order to achieve reliable performance of optical thin film systems such as filters, waveguides, optical MEMS, sensors, optical coatings on plastic substrates and others. Mechanical properties are closely related to the film microstructure and composition that, in turn, depend on the choice of materials and processes and on the energetic conditions during deposition. In order to assure successful performance and high stability of optical films, the mechanical properties have to be optimized together with the optical characteristics and other film functional properties (electrical conductivity, thermal and environmental stability, surface energy etc.). This presentation will give an overview of the methodology of mechanical testing and will describe the advantages and limitations of individual methods. We will concentrate on the effect of process parameters on the mechanical properties of the individual low, medium and high index optical films, especially those prepared by Physical Vapor Deposition (PVD) and Plasma Enhanced Chemical Vapor Deposition (PECVD). After a detailed review of the basic mechanical properties of films and substrates used in optical applications, we will illustrate the advances in this filed by examples of complex optical systems in which the control of the mechanical behavior is particularly important. This includes a comparison between inhomogeneous and multilayer films, optical coatings on plastics, complex optical filters used in optics, optoelectronics, photonics, architecture, and in medical, security, aerospace, automobile and other applications.

9:40am **TF-WeM5 Improved Optical Heterodyne Detected Transient Grating Method by using a Thin Film Grating**, ***K. Okamoto****, Z. Zhang,* California Institute of Technology*; D.T. Wei,* Wei & Assoc.*; A. Scherer,* California Institute of Technology

Transient grating (TG) spectroscopy based on the third order nonlinear optical effect has been applied to material, chemical, and biological research. Optical heterodyne detected (OHD) TG measurements have been reported by several groups, but experimental setups were mostly complicated. Here, we demonstrate a convenient new technique of OHDTG by transferring a pattern directly from a metal film grating into sample solution. This method has the same advantages of existing OHDTG techniques but the setting is much simpler. Thin film gratings having submicron periods are fabricated by: evaporating metal on a glass substrate, laser beam writing lithography, and chemical etching. The narrowest metal width of our grating is 333nm. The fabricated grating is then placed in the front side of a quartz cell containing the sample solution with 10mm optical pass length. An UV pump beam (a frequency-tripled Nd:YAG laser) casts a shadow from the grating to the sample solution such that a dark/bright pattern is formed in the solution liquid called transient grating. Such spatial modulation of the optical intensity induces changes of temperature, density and the molecular excitation. Through this transient grating, the modulated refractive index and absorbance of sample materials can be detected by the diffraction of a probe beam (a He-Ne laser). By analyzing the probe beam diffraction, we can obtain the intensity and dynamics of the modulated parameters named above. This technique has many advantages compared with existing OHDTG techniques; such as: (1) simple setting, easy alignment, (2) high signal stability, (3) easy control of phase shift, and (4) quick interchange of grating periods. We shall demonstrate this technique from several materials and discuss about the potential benefit of this new technique.

10:00am **TF-WeM6 Infrared Emission from Zinc Sulfide Doped with Rare Earth Fluorides**, ***D. DeVito****, A. Kale, W. Glass, M. Davidson, P.H. Holloway,* University of Florida

Thin film electroluminescent devices are excellent sources for high efficiency infrared emission. Rare earth elements, such as Erbium, Thulium and Dysprosium, are good dopants for infrared emitting phosphors because of the many transitions they exhibit in the infrared region. Zinc sulfide is a stable, wide bandgap semiconductor in which electrons can be excited to appropriate energies to produce excitation of the luminescent centers. Achieving good brightness requires a combination of precise composition, deposition conditions and post-deposition annealing. Lanthanide doped zinc sulfide electroluminescent thin films were deposited from two planar magnetron sources using ZnS and lanthanide trifluoride targets. Infrared emission from devices shows a marked improvement for films annealed at temperatures above 350°C. Maximum brightness was achieved for samples annealed at 425°C and, based on SIMS data, is associated with a reduction in the fluorine concentration in the films. Mechanisms for the loss of fluorine include solid state diffusion, with replacement of F with S on the substitutional site. The mechanism for increased electroluminescence will be discussed based on oscillator transition strength relative to nearest neighbor bonding.

10:20am **TF-WeM7 Spectrally Stable Optical Coatings Using Closed Field Reactive Magnetron Sputtering**, ***J.M. Walls****, D.G. Gibson,* Applied Multilayers Ltd, UK

Magnetron Sputtering has many advantages for the deposition of multilayer optical coatings. The sputtering process is "cold", making it suitable for use on the widest range of substrates including polymers. This paper will describe the "Closed Field" process that allows high quality, multilayer metal-oxide thin films to be deposited at high rates with exceptionally low absorption. In contrast to previous reactive dc sputtering strategies the Closed Field process does not require a separate ion or plasma source. The Closed Field creates a magnetic bottle that extends the electron mean free path leading to high ion current densities. The combination of high current densities with ion energies in the range 30eV to 50eV creates optimum thin film growth conditions. As a result the films are dense and spectrally stable. Also, they are exceptionally smooth as measured using FEGSEM and Coherence Correlation Interferometry Examples of the morphology and optical properties of single layer electrically insulating (SiO2, Nb2O5 and TiO2) and conducting meat oxides (ITO) will be presented. The structure and morphology of a series of multi-layer metal oxide coatings will also be presented These will include ant-

reflective coatings, UV and infra-red blockers, lighting filters and conductive oxides for flat screen displays.

10:40am **TF-WeM8 Multilayered SiN@sub x@/SiO@sub 2@ AR Coatings with Cylindrical Cathode by Unbalanced Compressive Magnetron Sputtering**, ***C.-S. Wang***, *K. Sasaki,* Kanazawa University, Japan; *S.-F. Chen,* National Taipei University of Technology, Taiwan; *T. Hata,* Center of Optical Coating Research, Japan

Excellent adhesion and high throughput production of anti-reflection (AR) coatings were examined by high and low refraction index SiN@sub x@/SiO@sub 2@ combined system. In general, the refraction of SiN@sub x@ was strongly dependent on the composition of nitrogen and it is difficult to obtain stoichiometric SiN@sub x@ in state-of-the-art techniques. In order to satisfy the requirements, we employed a cylindrical unbalanced compressive magnetron (UCM) sputtering technique. A pair of permanent magnets was adjacent to a cylindrical rotating target to increase and compress the ionization density in the region near the substrates. And the reactive gases were introduced between the cathode and UCM so as to produce discharge uniformly. We used a plasma-sprayed Si cylindrical target (6 cm in diameter and 50 cm in length) in which a 4.5 kGauss permanent magnet was built-in. Moreover, the same magnets were also sited to the both sides of the target to form convergence plasma stream forward to a glass substrate. The distance of target to substrate was 5 cm. SiN@sub x@ and SiO@sub 2@ films were deposited by alternatively changing N@sub 2@ and O@sub 2@ gases. The deposition rates of the films were as large as 2.01nm/s and 2.23nm/s respectively. The refraction index of SiN@sub x@ films was varied from 1.63 to 1.75 at 500 nm wavelength by varying Ar/N@sub 2@ flow ratio from 1 to 0, while that of SiO@sub 2@ films was 1.45 stably. According to AR simulation using above values, 4 layers system of SiN@sub x@/SiO@sub 2@ satisfies a reflection less than 1% in wide visible range (420~680 nm). Whole process without evacuation was completed within just 4 min. Additionally, according to spectroscopic ellipsometory measurement, the extinction coefficient was small resulted that high density and less defective films were obtained. This technique is useful for large scale and high performance AR coatings.

11:00am **TF-WeM9 Analysis of a Combined Sputtering and Plasma-Heating-Evaporation Process of Magnesium Fluoride by using Plasma Emission Spectroscopy and Energy Resolved Type Mass Spectrometry**, ***T. Deguchi***, Kanazawa Institute of Technology, Japan, Ishikawa; *T. Sasaki, E. Kusano,* Kanazawa Institute of Technology, Japan; *S. Iura, K. Kawamata,* Olympus Co., Ltd., Japan; *A. Kinbara,* Kanazawa Institute of Technology, Japan

A sputtering deposition process combined with plasma-heating-evaporation is an attractive technique to deposit MaF@sub 2@ thin films onto unheated substrate. However, poor understandings of the mechanisms prevent stabilization of the process. In this study, we have analyzed the process by using plasma emission spectroscopy and energy resolved type mass spectrometry. A sputtering machine used for mass spectrometry was a UHV system equipped with a 55 mm diameter cathode and that for film deposition and plasma emission spectroscopy was a commercial batch type system equipped with a 75 mm diameter cathode. The plasma process monitor (PPM-421, Balzers AG) was used to analyze a mass/charge ratio and energy of ions arriving to the substrate. A spectrometer (TYPE HR-2000, Ocean Optics) was used for plasma emission spectroscopy. Ions typically detected were Mg@super +@, F@super +@, MgF@super +@ both in Ar and O@sub 2@ atmospheres. While in Ar atmosphere the number of MgF@super +@ decreased with increasing rf plasma power, in O@sub 2@ atmosphere it increased drastically after the plasma power exceeded 250W. At this point in O@sub 2@ atmosphere the plasma-heating-evaporation occurred. Results obtained by the plasma emission spectroscopy well agreed to these results; i.e., the emission intensity ratio MgF@super *@/O@super *@ increased significantly at an rf power of 200W. Furthermore, the high energy tail in ion energy spectra, resulting from momentum transfer in sputtering process, observed for Mg@super +@ or F@super +@ in Ar atmosphere disappeared for those in O@SUB 2@ atmosphere, showing that the evaporation process in O@sub 2@ atmosphere was not dominated by sputtering, but by thermal evaporation.

11:20am **TF-WeM10 Silicon-rich Nitride Characterization for Polysilicon Gate Patterning**, ***F. Celii***, *K. Hewes, S. Zheng, E. Mickler,* Texas Instruments

For manufacturable patterning of sub-0.10 µm poly-Si gates, advanced etch techniques must complement current lithography capability. One approach uses photoresist line-narrowing combined with an etch hardmask that serves the dual purpose of forming a low-reflectivity patterning substrate. The hardmask suitability is determined by the poly-Si etch resistance, the optical properties (if used as an anti-reflection coating (ARC) layer) and integration issues (e.g., cleanup and cost). The requirements on the hardmask will change with subsequent generations, as the material stack layers and thicknesses change. We report the characterization of silicon-rich nitride (SRN) films used in poly-Si gate patterning. SRN films were deposited by PECVD in commercial reactors. Initial characterization used variable-angle spectroscopic ellipsometry (VASE), over the wavelength range 140 - 1100 nm. Modeling of the VASE data provides optical constants at lithography wavelengths (193, 248 nm) of the various film compositions. Based on previous work, the spectral dependence of the optical constants also provided a measure of the Si-loading of the film. The Si-content of the previously-studied sample set (37 to 41%),@super 3@ which gave k@sub 193@ values up to 1.0, was significantly extended with the current set, with k@sub 193@ values up to 2.0. Moreover, the (n,k) values of the current SRN films fall along a fairly narrow line. Additional characterization of these films will include composition measurement by RBS spectroscopy and FT-IR spectroscopy characterization over 400 - 4000 cm,@super -1@ which yield an independent measure of Si-dimer concentration. Simulations of gate patterning structures show (n,k) values which yield reasonable patterning process margin. CD swing curves of patterned films will also be presented.

11:40am **TF-WeM11 Film Stress and Discharge Properties of MgO protective layer of AC-PDP**, ***M.J. Lee***, *S.Y. Park, S.H. Moon, S.G. Kim,* Seoul National University, Korea; *H.J. Kim,* Seoul National University, Korea, South Korea

To increase the lifetime of PDP (Plasma Display Panel), MgO thin films are applied on dielectric layer to protect the dielectric layer from ion bombardments of plasma. Because MgO film is unlikely sputtered by ion bombardments and reduces the discharge voltage of PDP due to high secondary electron emission coefficient (@gamma@@sub i@), in turn less energy consumption. In this work, discharge characters of PDPs related to the stress formed in MgO thin films are investigated. MgO thin films were deposited on glass substrate with dielectric by e-beam evaporation in vacuum chamber, 5 x 10 @super -7@ Torr. Substrate temperature was varied from room temperature to 300°C, then annealing process is carried out at 300°C in vacuum chamber for 1hr and 3 hr respectively. The stresses of each sample were measured by laser scanning method to detect the substrate curvature change. To examine the effect of the change of stresses in MgO layers in different formation process on discharge character of real panel, firing voltage (V@sub f@) and sustaining voltage (V@sub s@) were observed. Surface roughness, morphology, and crystallographic preferred orientation of MgO films were also evaluated by XRD, AFM, SEM. And the impurities and hydrates of MgO surface before and after annealing were analyzed by XPS. MgO films on glass substrate have compressive stresses, which were varied according to deposition condition. After the annealing, the compressive stresses were released relatively, and definitely changed at specific temperature condition. The relation between the discharge characters and MgO thin film stress change and the relevancy with other properties of films was also evaluated.

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